# A Tribute to Professor David V. Edmonds on the Eve of his Retirement

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## Abstract

We highlight here the life and career of Professor David Vernon Edmonds, and his contributions to the study of microstructural evolution over the past four decades in Cambridge, Oxford and Leeds. The scientific discoveries in the Edmonds' group are highlighted in the context of microalloyed, ferritic, pearlitic, bainitic, and martensitic steels, as well as cementite and cast iron, and in a variety of non-ferrous fields including high-density tungsten and uranium alloys, titanium alloys and twin-roll thin-strip casting.

## Introduction

In celebrating the career of Professor David Edmonds, we recognize the central theme of his work on solid-state phase transformations and microstructural evolution involving the application of a variety of metallographic techniques, where he and his research group have made important contributions over four decades. David Edmonds was raised in London where he attended a local primary and grammar school before pursuing his B.Sc. (1962-1965) and Ph.D. (1965-1968) in Physical Metallurgy at the University of Birmingham. His doctoral studies on yield phenomena in zirconium and hydride embrittlement of titanium were with Jim Beevers, a former student of Professor Robert Honeycombe at Sheffield. This connection led him to join Professor Honeycombe's group at Cambridge, working initially on the interphase precipitation of particles at moving ferrite-austenite interfaces in alloy steels. The prestigious ICI Research Fellowship which funded his research was meant to support him for two years, but was followed by a succession of distinguished appointments resulting in 11 exceptional years at Cambridge (1968-1979). These included the British Steel Corporation Research Fellowship, and the Warren Research Fellowship of the Royal Society (previously held for many years by William Hume-Rothery) and a Fellowship at Corpus Christi College which carried responsibility for graduate students.

In 1979 David joined Oxford University in the Department of Metallurgy and Science of Materials (originally founded by Hume-Rothery in the 1950's) as a Lecturer in Industrial Metallurgy, and the first metallurgy Fellow at St. Anne's College. He was recruited to Oxford in part to help create the MEM program ("Metallurgy, Economics and Management"), designed to nurture captains of engineering industry. This involved an extra year of study, including a

management-training internship in industry, and remains today with a more fashionable title. In 1993, David was elevated to the Chair of Metallurgy and Materials, formerly held by Jack Nutting at the University of Leeds. This was a period of great change in the world of materials and David served two terms as the Head of Department over the periods 1995-1998 and 2001-2004, during which the activity metamorphosed into what is now the Institute for Materials Research, in the School of Process, Environmental and Materials Engineering at Leeds.

David's endeavours in science have been recognized most prominently by his election to the Royal Academy of Engineering and the Fellowship of ASM International. But there are many other marks of respect for his work, including the Vanadium Award from the Institute of Materials, Minerals and Mining in both 1993 and 2002. David has always given teaching a high priority. The Oxford "tutorial system" that he participated in is inherently hard work but enjoyable. Over the years, he has succeeded in explaining an impressive range of metallurgical subjects to students, including solid-state phase transformations, deformation, fracture, fatigue, creep, ferrous and other alloys, introductory materials science and engineering, along with a variety of processing-related aspects such as casting, working, welding, machining, surface finishing, heat treating.

But Cambridge is where David literally met his match. He married Jenny in 1970 in the Chapel at Corpus Christi College, to begin a lasting partnership, including the rearing of two children, Victoria and Alex, who were both christened at Corpus. Victoria has degrees from both Oxford (B.A. – biology) and Cambridge (Ph.D. - zoology) and is currently a British civil servant working in Whitehall, while Alex received his B.A. in Engineering Sciences (and rugby "Blue") from Oxford, and works in the structural engineering field. Family life has been an important value to the Edmonds, along with physical recreation. (David remains an avid jogger and competitive oarsman, perhaps explaining his excellent health and youthful appearance.) Dr. Jenny Edmonds had a parallel education and professional life, first meeting David at grammar school, receiving degrees from Birmingham, and holding appointments as a botanist at Cambridge, Oxford and Leeds, as well as the Royal Botanic Gardens, Kew. She was also a Fellow of Darwin College, Cambridge during a seminal period in the history of the College.

The United States of America has had special influence and importance in the life of David Edmonds. He has participated in many technical conferences in the USA, and often used personal time to extend his visits and develop strong relationships. David and Jenny Edmonds honeymooned during their first visit to America, and enjoyed sabbaticals at the Naval Postgraduate School in Monterey, California, where their children were exposed to the local culture and education. David is a Fellow of ASM International, so it is most fitting that this tribute takes place at the Materials Science and Technology conference. The authors of this tribute were privileged to have their doctoral research supervised by David during his years at Cambridge (HKDHB, 1976-1979) and Oxford (JGS, 1980-1983). In the sections that follow, we highlight some of the key developments attributed to the Edmonds' research group, focusing on the most stable element in the universe, iron.

#### **Research Contributions**

Recognising the importance of microstructure on the properties of metals, David has worked meticulously to characterise the evolution of detail in complex industrial steels.

#### Microalloying

Seminal work using transmission electron microscopy (TEM), begun at Sheffield and continued at Cambridge by Honeycombe and co-workers [1,2], had revealed two distinct forms of alloy carbide or carbonitride precipitation in ferrite. It seemed that tiny particles precipitated in arrays of parallel sheets, because they formed at austenite/ferrite transformation interfaces. This *interphase precipitation* and associated microstructures consisting of arrays of long fibres, was visually striking and remarkable in its organization (Figure 1). The arrays were found in the then emerging microalloyed steels and in heat-resistant power plant steels, and David was engaged by their effects on strength [3], creep [4,5], fracture [6] and fatigue [7,8]. His enthusiasm for high resolution microanalytical techniques was boosted by these newlydiscovered forms of precipitation, especially the fibrous form [9,10]. He pioneered the application of photoemission electron microscopy to look at the austenite as it decomposes [11-13], thus revealing for the first time, direct observations of the propagation of the transformation interface by a step mechanism, in a manner consistent with interphase precipitation [14]. This work led to a more general interest in austenite formation and decomposition, particularly in ways which circumvent the loss of parent austenite in partially transformed samples by martensitic transformation, an example being use of thin retained austenite films in the martensite [15] to relate the three-phase crystallography at the interface between parent austenite and product ferrite and 'interphase-precipitated' carbide and explain therefore the selection of particular crystallographic variants [16].





(a) (b) Figure 1 Electron micrographs of (a) interphase [3] and (b) fibrous precipitation [9] of alloy carbides in pro-eutectoid ferrite. Magnification is 35,000X (a) and 4,000X (b).

Another way to preserve austenite was with a high-Mn alloy such as Hadfield steel, which is typically austenitic at room temperature but can be in a state where cementite, ferrite and austenite are in three-phase equilibrium. This permitted the direct observation of the austenite/ferrite interface. In a microalloyed version, it helped to demonstrate the possibility of alloy carbides in *pearlitic ferrite*. This is of use in steels for forgings, rod, wire and rails [17-19], the precipitation occurring via an 'interphase' mechanism (Figure 2) [20,21]. Meanwhile, it became evident that microalloying elements can be exploited for applications other than grain refinement and precipitation strengthening. It was shown that minor amounts of V (and/or Si) can modify the initiation of austenite decomposition at the grain boundaries in high carbon rod steels (Figure 3) [22]. This mechanism was used to eliminate cementite films which embrittle grain boundaries in hypereutectoid steels, enabling the development of pearlitic wires stronger than 2350 MPa at practical diameters.



Figure 2 Interphase precipitation in pearlitic ferrite (dark-field electron micrograph) [16].

Figure 3 Electron micrographs illustrating modification of grain boundary cementite precipitation in a hypereutectoid steel (a) by a vanadium addition (b) [22].

#### Pearlite

In a program to study stress relaxation in spring steels, cold-drawn after patenting, atomprobe field ion microscopy (APFIM) was used to obtain one of the first direct observations of interstitial atom decoration of dislocation substructure (Figure 4) [23,24]. The dislocation substructure changed with low temperature heat treatment whereas the interstitial atmosphere was virtually unaffected.

Constant growth rate (forced velocity) experiments, instead of isothermal heat treatments, were also conducted on eutectoid and off-eutectoid steels [25-27] utilizing a steep temperature gradient to move a single transformation interface through the material at a controlled rate. These studies helped determine the diffusional transformation mechanism from the measured velocity/interlamellar spacing relationship, the extent of cooperative growth and the ability to align the lamellar microstructure; experiments were also extended to Cu-In alloys.



Figure 4 Imaging atom probe micrographs indicating carbon segregation to the dislocation substructure in cold-drawn eutectoid steel wire [24].

#### Bainite

The similarity between variant selection of interphase precipitation and that of cementite precipitation in lower bainite, and also the important experience of examining the ledged austenite/ferrite interfaces in reconstructive transformations, led to studies of the bainite transformation in the Edmonds group. At that time the bainite transformation appeared little understood as compared with, say, the martensite or pearlite transformations, which was partly responsible for less interest in the development of commercial bainitic steels. Thus began an interesting period exploring the bainite transformation mechanism, particularly with respect to its displacive or martensitic characteristics [28,29], and this work was followed by efforts to begin to develop a microstructural route to tough, high strength steels based upon bainite [30-36]. The experimental approach followed the seminal work of Hehemann and co-workers [37-39], using Si alloying to suppress cementite formation so that austenite decomposition to bainitic ferrite could be examined unhindered by the secondary reaction. The displacive mechanism remained controversial at the time [40-42] and to this day, but the work initiated a fairly wide and lengthy debate which at a minimum corrected the previous neglect of bainite as a useful microstructure and must in some way have contributed towards the subsequent emergence of TRIP-assisted steels [43-45] which are industrially important, and low-temperature transformed 'nanobainite' structures that have emerged more recently [46-47].

The new microstructural route to improved performance that has emerged is based upon *carbide-free* bainitic ferrite, brought about by the Si alloying addition (or Al additions in more recent studies). However, another microstructural feature is equally important, which is the presence of *interlath retained austenite*, stabilized by the unprecipitated carbon [48] (Figure 5). These two microstructural features provide the potential for mechanical property improvements: high strength, toughness, fatigue resistance and, because austenite might act as a sink for hydrogen as well as carbon, hydrogen embrittlement resistance. These concepts also hold for the newer Q&P steels [49-51], which essentially exchange martensitic ferrite for bainitic ferrite, but with more control over the carbon concentration of the retained austenite phase.





Figure 5 Electron micrographs of carbide-free bainite in a high-Si steel. Interlath retained austenite is revealed by internal stacking faults and dark-field imaging [48].

## Acicular Ferrite

With his interest in bainite and martensite, it was perhaps not unnatural that David turned his attention to a study of the so-called 'acicular ferrite' microstructure. This microstructure became a hot topic during the 1970's in the development of tough weld metals when a general consensus was emerging that intragranular nucleation at weld metal inclusions was responsible for the intragranular nucleation of acicular ferrite [52]. The Edmonds group contributed persuasive metallographic evidence for inclusion-assisted nucleation and sympathetic nucleation

(Figure 6(a)) along with the dependence upon inclusion content, austenite grain size and Mn concentration [53]. In the 1990's David returned to the topic, this time in order to explore the potential for developing this strong tough microstructure in *wrought steels*, and also to investigate the so-called "vanadium effect" whereby this element seemed to contribute towards an intragranularly-nucleated acicular ferrite structure [54-55]. Good metallographic evidence of the 'vanadium effect' was found (Figure 6(b)), but in this instance using high-purity laboratory-produced alloys, with an absence of any significant inclusion-assisted nucleation, and no visible vanadium carbonitride precipitation. High resolution techniques of scanning transmission electron microscopy coupled with energy dispersive X-ray analysis and secondary ion mass spectrometry indicated vanadium segregation within the microstructure, which may suggest that solute vanadium contributes to intragranular acicular ferrite formation via its interfacial or ferrite stabilizing effects.



(a)



(b)

Figure 6 (a) Electron metallographic evidence for inclusion-assisted intragranular nucleation of acicular ferrite in submerged arc weld metal [53], and (b) light micrographs illustrating influence of vanadium addition (bottom) promoting the acicular ferrite microstructure in 0.1wt% C steel [55].

#### **Austempered Ductile Iron**

The bainitic structures studied in steels alloyed with Si, led to an interest in austempered ductile cast irons, which, because of their similarly high Si content and austempering treatment possess equivalent carbide-free bainitic microstructures containing retained austenite. TEM studies by the Edmonds group characterized the bainite matrix which, although confirmed to be similar to that previously studied in steels, can contain segregation of the main alloying elements that influence final microstructure; Si, Ni and Cu graphitisers in the eutectic cell, and Mn, Mo carbide promoters in intercellular regions. The concentration gradients across the microstructure probably account for a number of carbides which were identified; cementite, epsilon, eta, kappa, and faulted triclinic carbide previously identified in high-Si steels [56,57]. Later studies concentrated on the effects of substituting aluminium for silicon, which resulted in a similar

matrix microstructure of carbide-free bainite with interlath retained austenite but also, importantly, enhanced oxidation resistance [58].

## Quenched and Partitioned (Q&P) Steels

Quenching and partitioning is a new concept for the heat treatment of martensite, somewhat different than customary quenching and tempering. The process involves quenching to below the martensite-start temperature and directly annealing, either at, or above, the initial quench temperature. If competing reactions, principally carbide precipitation, are suppressed by appropriate alloying, the carbon partitions from the supersaturated martensite phase to the untransformed austenite phase, thereby increasing its stability and retention upon cooling to room temperature (Figure 7). A key to successful heat treatment is prevention of carbide precipitation and so recent metallographic studies [59-62] have focused upon the conditions controlling precipitation of cementite and the transitional epsilon carbide, utilising mainly high resolution TEM but also the position-sensitive atom probe (PoSAP). David's group at Leeds has been instrumental in the characterization of Q&P microstructures, and particularly in helping to understand the transition carbide precipitation behavior in both low and medium carbon alloys subjected to Q&P processing. The importance of transition carbide solubility/stability has been critical in understanding how large quantities of austenite can be stabilized by carbon partitioning in some processing scenarios, while fine transition carbides are more common in others.



Figure 7 Bright- and darkfield electron micrographs of typical interwoven martensitic ferrite and retained austenite of Q&P microstructure [60].

# Graphitization

Solid-state nucleation and growth of graphite nodules in steels emerged from Edmonds' sustained interest in the effects of Si alloying combined with the more recent idea to explore the use of graphite as a route to improved machinability of steels, rather than additions of Pb, S, Te, Bi or P, which may impair cold forgeability, or create toxicity and environmental problems rendering the steel expensive and difficult to recycle. The equilibrium form of carbon in the Fe-C system is graphite, but kinetic considerations ensure that under normal heat treatments iron carbide (cementite) forms first. Long term annealing can replace cementite by graphite but is not a commercially-viable industrial process. However, by increasing the concentration of graphitizing elements, particularly Si, and reducing carbide forming elements, graphitizing times were significantly reduced to just a few hours [63-65]. Significantly, the experimental results, obtained by high resolution TEM and utilising a quantitative method of mapping Plasmon energy loss shifts within graphitizing carbon systems using electron spectroscopic imaging (Figure 8),

indicated that the refined dispersion of small graphite nodules nucleated from temper particles associated with cementite during the early stages of the graphitizing anneal [66-69]. Consistent with this hypothesis is the observation that graphitizing kinetics are influenced by the starting microstructure, either quenched martensite, austempered (high-Si) bainite or pearlite.



Figure 8 Bright-field TEM montage image and Plasmon Ratio Map indicating the less graphitic nature of the core compared with the outer layers of a graphite nodule about 2 microns in diameter [66].

## Proeutectoid Widmanstätten Cementite

The high-Mn Hadfield steels used to study features of the austenite/ferrite decomposition interface and interphase precipitation in pearlitic ferrite exhibit hypereutectoid behavior and presented an opportunity also to examine cementite precipitation in austenite [70] as well as another unexpected reaction discovered, the precipitation of elemental copper in cementite. Widmanstätten precipitation of proeutectoid cementite in hypereutectoid steels has not been studied extensively. However, the high-Mn alloy allowed a detailed study of its morphology, interface structure and orientation relationships to the parent austenite phase, and revealed a completely new orientation relationship [71], within 3° of  $(100)_C // (112)_A$ ,  $(010)_C // (02-1)_A$  and  $(001)_C // (-512)_A$ . Moreover, the morphology of the plates possessing this new orientation relationship [72] (Figure 9). These different Widmanstätten plates were subsequently shown also to occur in plain carbon steel, using retained austenite films in the martensite matrix to identify and confirm the new orientation relationship.

Elemental copper was found to precipitate in proeutectoid ferrite and pearlitic ferrite similarly to alloy carbides, and therefore by an assumed interphase precipitation mechanism. Unexpectedly, however, copper was also observed to precipitate in the high-Mn hypereutectoid alloys in *proeutectoid or pearlitic cementite* [73-77] (Figure 10). Besides opening up an interesting topic for more fundamental study (e.g. the copper can act as a marker of the progress of the transformation), this discovery may give the opportunity to improve the crack resistance of the cementite phase, and hence the fracture toughness of plain carbon steels and cast irons.

## **Anomalous or Irregular Microstructures**

*Ultra-low Carbon Steels* Developments in modern steelmaking techniques have allowed the control of carbon concentrations to low levels and the ferritic transformation microstructures at these carbon levels are sometimes not well understood. A systematic metallographic study of ferrite in ultra-low carbon iron alloys helps in classifying the microstructural forms and identifying potential mechanisms of formation as the steels tend towards pure iron and massive transformation [78].



(a) (b) Figure 9 Electron micrographs of proeutectoid Widmanstätten cementite plates in a high-Mn steel related to parent austenite by (a) the Pitsch orientation relationship, and (b) the Farooque/Edmonds orientation relationship [72].



Figure 10 Dark-field electron micrograph illustrating copper precipitation within the thinner lamellae of pearlitic cementite [77].

Abnormal Ferrite A less understood but well-documented phenomenon in high-carbon steels is that ferrite can form in *hypereutectoid* compositions decomposed at small undercoolings beneath the eutectoid temperature. Well-known to occur in carburised steels it has been termed "abnormal" ferrite. Observations have shown that this ferrite forms on the proeutectoid cementite which can continue to grow, thus suppressing the establishment of the 'cooperative growth' conditions required for pearlite, and a model for the carbon redistribution required for this behavior was developed [79] for this structure.

*Cr-steels* An unusual acicular morphology of ferrite/carbide aggregate in Cr-containing steels which forms in a temperature range between classical pearlite and classical bainite structures has been examined and attributed to a transformation mechanism analogous to that of inverse bainite, where ferrite coats proeutectoid cementite, but in this case, a chromium carbide phase [80].

#### **Steel Corrosion**

Although on the surface a departure from microstructural-based research, this work was conceived to investigate the resistance of more modern microalloyed steels to  $CO_2$  attack because this form of steel corrosion is the most prevalent form encountered in oil and gas production and transmission, where traditional carbon and low alloy steels have poor resistance, forcing alternative expensive solutions. The program paid special attention not only to steel alloying but also to microstructural variations resulting from different processing and heat treatment routes, and tested a number of microalloyed steels in simulated field environments. A five-fold improvement over X70 in  $CO_2$  corrosion resistance at less than 1.5 times cost was demonstrated (Figure 11) and, in particular, vanadium was identified as a useful microalloying addition in this environment [81].

Various additional contributions by the Edmonds group have been made to steel research in such areas as dual phase [82], martensitic [83] and austenitic steels [84].



Figure 11 Lower corrosion rates exhibited by experimental steels compared to standard X70 grade steel [81].

## **Non-Ferrous Alloys**

David Edmonds' research in steels for defence applications led to interest in non-ferrous alloys with high density for kinetic energy armor penetration. Impurity segregation to microstructural interfaces in liquid phase sintered tungsten heavy alloys, and precipitation of carbide and intermetallic phases (Figure 12), both of which reduce the mechanical properties, particularly toughness, were identified in W-Ni-Cu and W-Ni-Fe by TEM and early Auger spectroscopy [85-89]. Depleted uranium alloys provide an alternative high-density system, and three binary systems of uranium were studied by the Edmonds group: U-Ti, U-Mo and U-Nb. TEM and APFIM techniques characterized the martensite transformations [90,91], aging reactions and isothermal decomposition structures in these alloys [92,93], along with deformation microstructures [94]. The emerging PoSAP technique provided persuasive evidence for spinodal decomposition in U-6Nb (Figure 13), although still under discussion [95]. Also related to ballistic applications, complex deformation structures developed in FCC copper and



(b)

(a) Figure 12 (a) Light micrograph illustrating the microstructure of as-sintered W-Ni-Fe heavy alloy [86], and (b) electron micrograph of eta carbide identified at the tungsten/matrix interface [87].



(a)



Figure 13 (a) Three-dimensional PoSAP iso-concentration image, connecting regions containing 25at%Nb in aged U-6Nb, and (b) compositional map sliced from three-dimensional image, indicating percolated nature of the microstructure [93].

BCC iron over the strain-rate range  $10^3$  to  $>10^{-5}$  s<sup>-1</sup>, achieved by Hopkinson bar tests and explosively forming projectiles, were examined by TEM [96]. Ultra-high strain rate deformation is not well understood, although important in modeling and understanding ordnance applications, explosions, high-speed impacts and earthquakes.

The use of hydrogen as a temporary alloying element during fabrication of Ti-matrix composites and heat treatment of Ti<sub>3</sub>Al based aluminides was explored. Hydrogen is a betastabilizer and prehydrogenation and postdehydrogenation treatment lowers the phase transformation temperatures, and hence fabrication and heat treatment temperatures, to give less interface degredation between fibre and matrix in Ti-based metal-matrix composites [97] as well as refined microstructure in the titanium aluminides [98,99].

An important departure from microstructural investigations during Prof. Edmonds' career was to join with Prof. John Hunt, FRS in a joint industry-university initiative to upgrade the performance of the twin-roll thin-strip casting process [100-103]. Features limiting high-speed thin-strip casting such as heat lines, sticking, buckling and segregation were investigated on laboratory-scale casters (Figure 14), and aluminum alloys were cast at thinner gauges and higher productivities than were customary with commercial machines, enabling the revolutionary design and installation of twin-roll casters (*Davy Fastcast*) in two European plants. These facilities cast 10 ton coils at speeds up to 10 m min<sup>-1</sup> and thicknesses down to 1 mm in AA 8xxx series alloys.





Figure 14 Laboratory experimental twin-roll casters.

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